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फेरो क्रोमियम का रासायनिक विश्लेषण

भाग 6 निम्न कार्बन फेरो क्रोमियम में क्रोमियम का निर्धारण

Indian Standard

CHEMICAL ANALYSIS OF FERROCHROMIUM

PART 6 DETERMINATION OF CHROMIUM IN LOW CARBON FERROCHROMIUM

ICS 77.100, 71.040.40

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1997

Price Group 1

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 6) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferroalloys' covers the chemical analysis of different ferroalloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard, the Sectional Committee had decided that a separate series for each ferroalloy be prepared. Accordingly, IS 1559 in its various parts was published for the chemical analysis of ferrosilicon.

The chemcial analysis of ferromolybdenum is covered in a series of parts of IS 12614. The chemical analysis of ferrochromium is covered in a series of parts of IS 13452. With the publication of these parts the analysis of relevant constituents prescribed for ferrochromium in IS 1559 : 1961 will be superseded. This part covers the method for determination of chromium in low carbon ferrochromium. The other parts are :

Part 1 Determination of silicon in high carbon ferrochromium
Part 2 Determination of silicon in low carbon ferrochromium
Part 3 Determination of phosphorus in low carbon ferrochromium
Part 4 Determination of total sulphur in low and high carbon ferrochromium
Part 5 Determination of chromium in high carbon ferrochromium
Part 7 Determination of nitrogen in high and low carbon ferrochromium
Part 8 Determination of carbon (total) in low and high carbon ferrochromium

In this revision the determination of chromium in low carbon ferrochromium has been updated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off vlaue should be same as that of the specified value in this standard.

Indian Standard

CHEMICAL ANALYSIS OF FERROCHROMIUM

PART 6 DETERMINATION OF CHROMIUM IN LOW CARBON FERROCHROMIUM

1 SCOPE

This standard (Part 6) covers the method for determination of chromium in the range of 60 to 70 percent in low carbon ferrochromium, containing carbon up to 0.1 percent.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
1070 : 1992	Reagent grade water (*third revision*)
1472 : 1977	Methods of sampling of ferro alloys for determination of chemical composition (*first revision*)

3 SAMPLING

Sample shall be drawn and prepared in accordance with IS 1472.

4 QUALITY OF REAGENTS

Unless specified otherwise, reagent grade water (*see* IS 1070) shall be employed for test.

5 DETERMINATION OF CHROMIUM IN FERROCHROMIUM (CONTAINING LESS THAN 4 PERCENT SILICON) BY PERSULPHATE OXIDATION METHOD

5.1 Outline of the Method

After dissolution of the sample in a dilute sulphuric acid and phosphoric acid mixture and further treatment with nitric acid, chromium, manganese and vanadium (if present) are oxidized by ammonium persulphate in presence of silver nitrate as catalyst. Permanganic acid is then destroyed by dilute hydrochloric acid. Chromium is reduced by ferrous ammonium sulphate and excess of ferrous ammonium sulphate is back titrated with standard potassium permanganate solution.

5.2 Reagents

5.2.1 *Phosphoric Acid — Sulphuric Acid Mixture*

To 600 ml of water, add cautiously 200 ml of sulphuric acid (rd = 1.84) and 100 ml of phosphoric acid (rd = 1.75). Mix, cool and dilute to 1 litre.

5.2.2 *Concentrated Nitric Acid*, rd = 1.42 (Conforming to IS 264).

5.2.3 *Silver Nitrate Solution*, 1 percent.

5.2.3.1 Dissolve 10 g of silver nitrate crystals in water and dilute to 1 litre.

5.2.4 *Ammonium Persulphate Solution*, solid.

5.2.5 *Dilute Hydrochloric Acid*, 25 percent (v/v).

5.2.6 *Dilute Sulphuric Acid*, 5 percent (v/v).

5.2.7 *Standard Potassium Permanganate Solution*, 0.1 N.

5.2.7.1 Dissolve 3.2 g of potassium permanganate in 1 litre of water. Let it stand in the dark for 2 weeks. Filter without washing, through a gooch crucible or a fine porosity fritted glass crucible. Avoid contact with rubber or other organic material. Store in a dark coloured glass stoppered bottle.

5.2.7.2 Dry a portion of sodium oxalate at 105 ± 5°C. Transfer 0.300 g of sodium oxalate to a 600 ml beaker. Add 200 ml of dilute sulphuric acid (5 percent) (previously boiled for 10 to 15 minutes and cooled) and heat to dissolve the oxalate. Titrate the hot oxalate solution at about 60 to 70°C with permanganate solution until a faint permanent pink colour persists for 30 seconds. Towards the end of titration one should take care to allow each drop to become decolourised before adding the next drop. To determine the blank, titrate 200 ml of dilute hydrochloric acid (25 percent) treat as above with potassium permanganate solution to a faint pink colour. The blank correction is usually equivalent to 0.03/0.05 ml.

Calculate the normality of potassium permanganate solution.

5.2.8 Ferrous Ammonium Sulphate Solution, (0.1 N approximate) Dissolve 39.2 g of ferrous ammonium sulphate in 1 litre of sulphuric acid (5 percent).

5.2.9 *Phosphoric acid* , 20 percent (v/v).

5.3 Procedure

5.3.1 Weigh accurately about 0.500 g of the sample into a 400 ml beaker and add 25 to 30 ml of phosphoric acid and sulphuric acid mixture and keep it for dissolution on a hot plate. Heat until effervescence nearly ceases. Add cautiously 3 to 5 ml of concentrated nitric acid and evaporate to fumes. Cool the solution, dilute to about 100 ml and warm to dissolve salts. Transfer the solution to a 250 ml volumetric flask and make up the volume with water.

NOTE — Chromium forms sparingly soluble salts in the presence of nitric acid and concentrated sulphuric acid. It is, therefore, necessary to avoid excessive fuming.

5.3.2 Take 50 ml of aliquot (**5.3.1**) containing 0.1 g of sample and dilute to about 200 ml. Keep the solution for boiling on a hot plate, keeping glass rod in the beaker to prevent bumping at a later stage. While boiling add 20 ml of silver nitrate solution and add 4 to 5 drops of potassium permanganate (25g/l) and then add 25 ml of freshly prepared ammonium persulphates solution, (20 percent). Boil the solution for about 15 to 20 minutes. If a permanganate colour due to the presence of manganese in the sample does not develop within a few minutes add more silver-nitrate and ammonium persulphate solution and boil again for 10 to 15 minutes to decompose the excess of persulphate. To the boiling solution add 10 ml of dilute hydrochloric acid (25 percent) in small portions of 2 ml and continue boiling till permanganate colour disappear.

In case either permanganate colour or any precipitated manganese dioxide do not disappear, add a little more of dilute hydrochloric acid (25 percent) and boil as before.

NOTE— During the process of boiling care should be taken to avoid losses of water by evaporation by adding water from time to time and thus always maintaining the original volume of the solution.

5.3.3 Cool the solution (**5.3.2**). Add a known excess of ferrous ammonium sulphate solution (0.1 *N* approximate) and 10 ml of phosphoric acid (20 percent). Stir and titrate the excess of ferrous ammonium sulphate solution with standard potassium permanganate solution (0.1 N) until a faint pink colour is obtained (V_2).

5.3.4 *Blank*

Boil the solution, till the permanganate colour is destroyed. Cool and add the same volume of ferrous ammonium sulphate as used for the sample. Titrate with standard potassium permanganate solution (0.1 N) to the appearance of pink colour (V_1).

6 CALCULATIONS

Calculate the chromium content as follows:

Chromium, percent by mass)

$$= \frac{(V_1 - V_2) \times N \times 0.01734 \times 100}{m}$$

where

V_1 = volume in ml, of standard permanganate required by the blank;

V_2 = volume in ml, of standard permanganate required in the experiment;

N = normality of the standard permanganate solution; and

m = mass in g, of the sample in the aliquot taken.

7 DETERMINATION OF CHROMIUM IN FERROCHROMIUM (CONTAINING MORE THAN 4 PERCENT SILICON) BY FUSION METHOD

7.1 Outline of the Method

After fusion of the sample with sodium peroxide and sodium carbonate instead of sulphuric acid and phosphoric acid same method is followed as given in **5.1**.

7.2 Reagents

All the reagents given in **5.2.3** to **5.2.9** are used along with the following:

7.2.1 *Sodium Peroxide*, solid.

7.2.2 *Sodium Carbonate*, Anhydrous.

7.3 Procedure

7.3.1 Transfer 0.5 g of the sample to a nickel crucible and add 8 g of sodium peroxide and 4 g of sodium carbonate and mix intimately. Cover the crucible and warm gently for about 5 minutes. Fuse the mixture over a low flame while imparting a swirling motion to the crucible. After holding molten mass for three minutes, increase the temperature to bright red for a minute and allow to cool.

7.3.2 Transfer the crucible and the fused mass to a 100 ml beaker, add hot water until the crucible is just submerged and heat gently until the melt is extracted. Remove the crucible and lid, washing with hot water and boil the solution slightly. Cautiously acidify with dilute sulphuric acid (25 percent) and add 50 ml in excess. Add 20 ml of phosphoric acid (20 percent), cool and make up volume to 250 ml with distilled water in volumetric flask.

7.3.3 Take 50 ml of aliquot (**7.3.2**) containing 0.1 g of sample, in a 400 ml beaker and complete the determination following the same procedure as specified in steps **5.3.2** to **5.3.4** and calculate the percentage of chromium as given in **6**.

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